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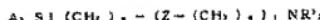
(54) AQUEOUS SILICONE EMULSION WHICH FORMS SILICONE ELASTOMER HAVING IMPROVED ADHESION TO SUBSTRATE

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain an aqueous silicone emulsion having improved storage stability and adhesion by mixing water with a surfactant, a crosslinking agent, a tin condensation catalyst, a specified diorganosiloxane polymer, a specified amino-functional siloxane and an acid.



SOLUTION: A diorganosiloxane polymer represented by formula I [wherein (n) is 0-3; (z) is 200-15; X is hydroxyl or a hydrolyzable group; R is a 1-5C (un)substituted monovalent hydrocarbon group; R1s are each X or R, provided that at least 90% of them are R; Y is Si, -Si-(CH₂)_mSiR12-group, -Si-(CH₂)_m-SiR12-SiR12-(CH₂)_mSiR12-group and (m) is a positive integer] is mixed with water, a surfactant, a crosslinking agent, a tin condensation catalyst, an amino-functional siloxane prepared by reacting an amino-functional silane represented by formula II [wherein A is a hydrolyzable group; Z is O or NR₂; R2 is H or a 1-15C (un)substituted monovalent hydrocarbon group; (p) and (q) are each 2-10; and (r) is 0-3] with a hydroxyl-terminated organosiloxane represented by formula III (wherein R4 is R; and (b) is 4-80) and an acid.



I



II

III

* NOTICES *

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2.**** shows the word which can not be translated.

3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
 [Claim 1] ORUGANO silicone polymer (1) which is a drainage system silicone emulsion which forms a silicone elastomer which has an improved adhesive property to substrate when water is removed, and is shown with a silicone emulsion(A) following formula containing the following
 $X_3 R_1 R_2 R_3 SiO_2 - Y - R' X_1 - SiO_2 - Z - Y - R' X_1 - SiO_2 -$ — here, n is 0, 1, 2, or 3, and z is an integer of 200-10000, X is a hydronium SHH(R) group or a certain hydroxyl basis, and R individually, 1-15 carbon atoms were replaced, or it is an unsubstituted universal hydrocarbon group, R' is individually chosen from X basis and R group, however at least 80% of R' is a group Y And an Si atom and -Si-(CH₂)_mSiR'₂-basis, Or (B) water whose m is -Si-(CH₂)_mSiR'₁ 2-O-SiR'₂ 2-(CH₂)_mSiR'₂-basis, and is as having

defined R' here above, and is a positive integer;

(C) A surface-active agent;

(D) Arbitrary cross linking agents;

(E) A tin condensation catalyst;

(F) Amino functionality Silane (1) shown with a following formula

$A_3 Si(CH_2)_p - O - (CH_2)_q - NR^3 - Si(CH_2)_p - O - (CH_2)_q - NR^2$ (here) A is a hydroxyl basis and Z is an oxygen atom or NR² (chosen out of a univalent hydrocarbon group by which a hydrogen atom and 1-15 carbon atoms were replaced individually, or R² is not replaced here), R¹ is chosen from a univalent hydrocarbon group which a hydrogen atom and 1-15 carbon atoms were replaced individually, or is not replaced, and p and q are the positive integers of 2-10, respectively — r — an integer of 0-3 — it — with an included ingredient, Formula HO-(SiR'₂)_b OH (R'₂ is chosen from a univalent hydrocarbon group which 1-15 carbon atoms were replaced individually, or is not replaced here, and) b — a positive integer of 4-80 — it is — effective dose of amino functionality siloxanes; formed by making hydroxy end ORGANO silicone (1) bloom react, and (G) — arbitrary acid.

[Translation done.]

IB 001-183302 A [DETAIL ED DESCRIPTION]

[0015] R and R¹ are a methyl group or a phenyl group, and a phenyl group preferably, R and R¹ is a phenyl group much more preferably.

[0016] The substituent S is single silicon atom (Si) or formulae-S-[CH₂]_mSiR¹- or -Si-(CH₂)_mSiR¹-2-0-SiR¹-2-[CH₂]_mSiR¹-2- (herein R¹ is as having given the definition above — m — a positive integer — it is — a base containing a methylene spacer group combined with silicon shown is expressed by Y is a silicon atom more preferably for this invention.

[0017] As long as it averages four molecules and at least 1/3 hydroxyl, hydroxyl bases, or these mixtures exist, said JORUGANO siloxane polymer may exist as a mixture of different JORUGANO siloxane polymer. Preferably, average over one molecule of JORUGANO siloxane polymer, and at least 1/3 hydroxyl, hydroxyl bases, or these mixtures exist, and most preferably, it averages per one molecule of polymer, and at least two hydroxyl, hydroxyl bases, or these mixtures exist.

JORUGANO siloxane polymer which exists as a mixture is prepared mixing two or more individual JORUGANO siloxane polymer before encapsulation, or by mixing an emulsion which encapsulated them individually and subsequently these emulsions are destabilized.

[0018] An example of desirable JORUGANO siloxane polymer, hydroxyl/ terminal PDMS polymer

and ethylene — dörin — there are RUKOGANO silv and PDMS polymer and ethylene dialkoxy alkyl and PDMS polymer. Here, as an example of a desirable alkoy group, it is methyl as an example of methoxy and thence with ethoxy ***, and a desirable aldy group. Hydroxyl terminal PDMS polymer is more desirable JUROGANO siloxane polymer.

can also be added at the time of versatility of a wash or a combination process. For example, in a desirable method of using emulsification directly, a JORIGANO[®] silicone and an ingredient of a wash of either are emulsified using water of two to five weight section on the basis of a small amount of water, for example, JORIGANO[®] silicone polymer 100 weight section. Subsequently, an obtained emulsion is diluted with additional water to a polymer solid content of a wash.

An ingredient (O) is a surface-active agent. A term "surfactive-active agent" describes a surface active agent chosen from a cationic surfactant, an anionic surfactant, an amphoteric surface active agent, nonionic surface active agents, and their mixtures, and has a dispersing phase of an emulsion. Although each of those surface-active agents will have stabilized individually, it will be combined with another surface-active agent of other kinds, and it will be added, but it is publicly known that it is difficult to stabilize an emulsion of DIOURANO (alcanoane sulfonate) by itself. Although an emulsion of a suitable olefino surfactant, fatty amines, and their derivatives, for example, dodecyl amine acetate, octadecyl amine acetate, and acetate of amine of behenyl fatty acid, is stable, it is not stable enough.

(dodecanoylamine). Aliphatic series amide derived from aliphatic dicarboxylic acids. For example, undecanedioylamine; A PAH derivative; quaternary ammonium compound which is ethylenediamine. For example, chloridation beef tallow trimethylammonium, diisooctyl chloride dimethylammonium. An amide derivative of amino alcohol, for example, beta-hydroxyethyl stearamide; — amine salt of 8 carb chain fatty acid]; — e quaternary ammonium base derived from fatty amine of 2 substitution diamine. For example, oleyl trimethylammonium and dihexadecyl chloride dimethylammonium. A quaternary ammonium base of hexadecylamine hydrochloride. A quaternary ammonium base of heptadecylamine hydrochloride. A quaternary ammonium base of behenylamine hydrochloride. For example, methyldiphenylbenzeneborate; bromate; pyridine, a basic compound of the derivative. For example, cetyl pyridinium chloride. A sulfonium compound, for example, a quaternary ammonium compound of an octadecyl aluminon methyl sulfate, betaine, for example, a betaine compound of diethylenetriamine acetic acid or octadecyl chitosan; ether; there are urethane [of ethylenediamine], and condensation product; polyethylenimine [of ethylenediamine], and polypropenal polyethyleneimine.

[Benzoyl ester of alkaline metal salt of sulfonate of coconut oil acid. Sulfonation of unsaturated alcanoic ester, for example,]
oleylbenzoate. Anide of aminosulfonic acid, for example, the oily **MECHIRUTA** reverse side —
 sodium salt [of C_8] — sulfonation output of fatty acid anion for example, palmitinato sulfonate.
Sulfonation aromatic hydrocarbon. For example, condensation product octahydronaphthalene sulfone sodium which it is with this sodium, naphthalene sulfone acid which is alpha-raphisthalene mono-
 sulfone, sodium and dibutylidodecylbenzenesulfonic acid (DBSA), and formamide.
Alkaline metal alkyl sulfates. For example, sodium lauryl sulfate; which has eight carbon atoms or
 example, there is a suitable amphotrophic surface active agent, that are leolin, bycantine,
 betaine, Sora Taine (lauretin), and alkylene propionate. At these examples, coco-ampholyte
 (coco-amphibetaine), (coco-amphocarboxy) glycerin (coco-amphocarboxy-butyrate). There are
 also — diethylaminoethyl sodium, and sodium lauryl sulfate. For example, cocamidopropyl
 betaine, and cocamidopropylamine (cocamidopropylbetaine).
SURFACTIN. An organic compound consisting of a long-chain amino acid, a long-chain
 alcohol, and a long-chain carboxylic acid. It is a surfactant, and is used as a surfactant in cosmetics.

[0025] As an example of a useable nonionic polyoxyalkylene solubilizer, polyoxyethylene ester, polyoxyethylene ether, polyoxyethylene amide, an ethoxylation sulfone, or a polyoxypropane oxide, etc. ether, ethoxylated amine, an ethoxylation sulfone and a nonionic surface active agent are preferred. A nonionic surface active agent, for example, a mixture of *tergitol* (trademark) [0026] (Amidol) X-10 and Triton X-100, and a mixture of *teritol* (trademark) X-10 and a nonionic surfactant, for example, laurofatty acid diacetyle, and a nonionic surface active agent is more preferred.

on the basis of JORUGANO silicone polymer 100 weight section. Quantity of a surface-active agent is 1.5 to weight section on the basis of JORUGANO silicone polymer 100 weight section more preferably.

JP-09-183902A [DETAILED DESCRIPTION]

JIORUGANO siloxane polymer. When this JIORUGANO siloxane polymer has a basis of many condensation nature or hydrolysis nature from an average of 1.3 per molecule, a cross linking agent for connecting a bridge in this JIORUGANO siloxane polymer is not required. A basis of condensation nature on a different JIORUGANO siloxane polymer molecule or hydrolysis nature forms a rubber particle which constrictured the bridge in response to mutual, and it will dry, if water is removed from said emulsion, and then forms a silicone elastomer. Probably, it will be difficult to use, though a cross linking agent is not required.

[0028] When said JIORUGANO siloxane polymer has a basis of many condensation nature or hydrolysis nature from an average of 1.3 per molecule, in order to form a rubber particle which will dry if water is removed from said emulsion, and forms a silicone elastomer, a cross linking agent must be added.

[0029] Typically, said cross linking agent is an organic silicon compound or resin containing a hydrolytic basis or hydroxyl which was averaged and was combined with many silicones from two or more molecules. A hydrolytic basis useful as a cross linking agent is the same as having described the substituent X of JIORUGANO siloxane polymer previously.

[0030] Said cross linking agent may also combine a basis of other kinds with silicones that combined with JIORUGANO siloxane polymer, as long as it has those bases that combined preferably with at least three silicon hydroxyls, basis combined with much silicon from an average of two places, or hydroxyl. Molecular structure of this cross linking agent is the Siang structure or siloxane structure. A siloxane type cross linking agent may be a straight chain, branched chain, or amine. A hydrocarbon group which univalent, which indicated a nitrogen atom and R] was replaced as an example of a basis bases hydroxyls Motosomchi who has combined with a silicon atom of said cross linking agent, and is not replaced is included.

[0031] Short, any publicly known cross linking agents which construct a bridge in a dielation system or siloxane emulsion under existence of a tin condensation catalyst can use them for this invention, and as the example, there are colloidal silica, alkali silicate, alkali siloxane, alkoxysilane, and ARIKEKOKI gardenia fruit ran, an OMSHMO silane, silanezene a friend NOKISHI silane, silicone resin, and a silicon hydride. As an example, a low-molecular-weight or spinach, silicone for example, a polymeric hydrogen siloxane, A low-molecular-weight copolymer which has a methyl hydrogen siloxane and a dimethyl siloxyl group, -OSi(OEt)₂-, -cyclohexylsiloxane, and tetramethyl orthosilicate, isobutyl trimetoxysilane, MICHIRUTORI propenoxysilane, vinylmethacryloxysilane, There is vinyl tri KISHIMO silane and Me₂SiOMe₂SiO₃ (MeON (Et)₂)² SiO₃ in such case, Me is methyl and Et is ethyl.

[0032] Said cross linking agent is Silox which has at least three siloxy groups. A time of each siloxy group having a carbon atom up to eight pieces is more prefer. A time of each siloxy group having three carbon atoms is the most preferable, for example, it is a time of being methyl trinonylsilane, isobutyl trimetoxysilane, methyl trihexylsilane, and MICHIRUTORI propenoxysilane, and chlorosilyl trimetoxysilane.

[0033] Typically, a cross linking agent is added by 0.1 to 50 weight section on the basis of JIORUGANO siloxane polymer, 100 weight section, when used by this invention, it is preferably added by one to 10 weight section per JIORUGANO siloxane polymer 100 weight section, and is added by two to 5 weight section per JIORUGANO siloxane polymer 100 weight section still more preferable.

[0034] Cross linking agent is added as a single kind or a mixture of a time of a different kind beyond 2 or 3. This cross linking agent can be added before emulsifying to the batch; however, it is preferred to add from an ease of combination, before emulsifying a cross linking agent.

[0035] An ingredient (E) is a tin condensation catalyst which is between this polymer and JIORUGANO siloxane polymer has sufficient hydrolytic basis, or is useful to promote crosslinking reaction between JIORUGANO siloxane polymer and a cross linking agent more typically. This catalyst is an organic tin compound of the valence+4, for example, 2 and tin (IV) compound, and an organo tin compound of tin (IV) and the valence+2, for example, a mixture with a 1st tin (II) compound, A 2nd tin (IV) compound is preferred. As an example, in the 1st tin, the 1st tin of octaneoic acid, the 1st tin of oleic acid of carboxylic acid organic, for example, the 1st tin, the 1st tin of octaneoic acid, and the 1st tin of neo dicarboxylic acid.

SUTANNOKISAKI (tin stanonoxane) — for example [(C₄H₉)₂SnCl]₂O and (C₄H₉)₂SnCl-O-Sn(OH)(C₂H₅)₂. Or there is formula R₁Sn(OCOR')₂ (R' is individually chosen from a univalent allyl group with 1-15 carbon atoms here, and R' is R or -CH₂GOR individually chosen from R is defined here above).

a tin catalyst — R'₂Sn(OAc)₂, (a) R'Sn(OCOR')₂ — (b) Sn(OCOR')₂, the dicarboxylic anhydride'; (c) carboxylic acid; (d) 2nd amine, or (N—(2-aminobutyl) amine, (e) Sn(SiR)₄—here) a is 1, 2, or 3; it may be a resultant (f) carboxylic acid, alcohol, or alcohol amine, A, tin (IV) compound is formula R'₂Sn(SiR)₂—D or R'₂SnD₂ (here), R' as having given the definition above and D is diecarboxylic acid monosester, c is certain-M-Sn(R')₂-OSi(R')₂-X-M' (here) at a positive integer of 1-10. M may be an allylic group on tin, and other bases that can be coordinated, and x may be a compound with certain-X, or R'₂Sn(SiR')₂ by a positive integer of 1-10.

[0036] Substituent R' is a univalent allyl group with 1-18 carbon atoms. As an example of R', there are methyl, ethyl, propyl, hexyl, diisopropyl, and octadecyl.

[0037] The substituent R is the dicarboxylic acid monosester (ethyl oxolato). For example, an ethyl o xoate.

[0038] The substituents M are an allyoxy group on tin, and other bases that may be coordinated, for example, acetylacetone.

[0039] Tin (II) carboxylic acid is a more desirable tin condensation catalyst, and dibutyltin diacetate is the most preferred.

[0040] In a silicone emulsion of this invention, it is sometimes desirable to reduce quantity of tin (IV). Coexisting with this publicly known, for example, Chu, H.K., Crosa, R.P., and Chasan, D.L.J. Organo-Chem. carbonyl organic, which indicated to 1992,259-17, or emuls. And it is attained by using a salt of carboxylic acid or amine.

[0041] A tin condensation catalyst is added in quantity of 0.01 to 1 weight section on the basis of JIORUGANO siloxane polymer 100 weight section. Preferably, this catalyst is added in quantity of 0.05 per JIORUGANO siloxane polymer 100 weight section — the amount part of duplex, and is added in quantity of 0.05 to 0.5 weight section per JIORUGANO siloxane polymer 100 weight section still more preferably.

[0042] A tin catalyst is a single kind or is added as a mixture of a different kind beyond 2 or it. The tin condensation catalyst can also add a front start of emulsification behind. However, as for a tin catalyst, adding before emulsification is preferred because of an ease of combination.

[0043] An ingredient (F) is indicated to be by an emulsion containing amino functionality Silox (II) an ingredient (F) is indicated to be by A₃Si(CH₂)₂-P(Z-(OH-CH₂)-N(R')₂; it is an amino functionality siloxane formed by masking hydroxy end ORGANO alkoxane (III) shown by formula HO-(SR')₂-OH react. Both and amino functionality Silox available.

[0044] A tin catalyst A is a hydrolytic basis, A, hydrolytic basis also includes a basis of a sap to combine with silicon hydroxyl with water at a room temperature. A suitable hydrolytic basis shown which hydrogen atom and 1-15 carbon atoms were replaced individually, or is not replaced here).

[0045] The substituent Z is an alkoy group which has a carbon atom to 8 more preferably. As an example of an alkoy group, there are methoxy and ethoxy propoxy, isobutoxy, pentoxy, HEKSOKSHL and 2-ethyl HEKSOKSHL An alkoy group has a carbon atom to 3 as A is the most preferred.

[0046] The substituent L is an oxygen atom or NR₂ (R' is chosen from a univalent hydrocarbon group which hydrogen atom and 1-15 carbon atoms were replaced individually, or is not replaced here).

The substituent Z is NR₂ preferably, R₂ was replaced or an example of a univalent hydrocarbon group which is not replaced is the same as a case of the substituent R. R₂ is a hydrogen preferably, which is not replaced.

[0047] Inferior-letter Q is a positive integer of 2-10, is 2 or more preferably, and is 3 most preferably.

[0048] Inferior-letter Q is a positive integer of 2-10, is 2 or more preferably, and is 2 most preferably.

[0049] Inferior-letter Q is an integer of 1-3, is 2 or more preferably, and is 0 or 1 most preferably.

[0050] Substituent R' is individually chosen from a univalent hydrocarbon group which 1-15 carbon atoms were replaced, or is not replaced. An example of these univalent hydrocarbon groups that is a acid, and the 1st tin of neo dicarboxylic acid.

IGCC 10000A [DETAILED DESCRIPTION]

[0045] Hydroxy and ORGANO siloxane of a short chain is more preferred than a long chain. That is because concentration of an affective amino functional group will be obtained with a comparatively small amount of an amino functionality siloxane if a short chain is used. It is because character of optimal chain length and a hydroxy and ORGANO siloxane is determined by parameter of one key parameter, compatibility with other ingredients of JIORGANO siloxane polymer of one another. One key parameter is compatibility of a hydroxy and ORGANO siloxane polymer of one amino functionality siloxane, and an emulsion. Compatibility improves with chain length in whom a hydroxy and ORGANO siloxane increases, and the chemical similarity of various R substituents. For example, if a JIORGANO siloxane is PDMS polymer substantially, it will be preferred to use a short chain PDMS for improved compatibility. One with important compatibility is polymer, and it contains a portion of aqueous phase, a discrete phase containing JIORGANO siloxane in a hydrophilic (H) in a deirable mode, said amino functionality siloxane is formed by making Silanol (IV) shown by amino functionality Silanol (III), hydroxy and ORGANO siloxane and, $R^5_2SiO_2$ react. It is

commercially available or this Slang is perceived by a public not given method. [0015] Substituent R¹ is chosen from a univalent hydrocarbon group which 1-15 carbon atoms were replaced individually, or is not replaced. An example of these bases is the same as what was given about the substituent R. Each R¹ is meantly preferable.

[0016] The substituent G is a basis of hydrolysis nature. A basis of hydrolysis nature useful for this invention is the same as having described X. The substituent G is an alkoxy group preferably and is methoxy, ethoxy, butyl more preferable.

[enriched] inferior letter c is 0, 1 or 2 is 0 or 1 and c is 1 more preferably, 0.005. An example of Silan (N) is the same as an example given towards, not only about dimethylmethoxysilane, dimethyl dichlorosilane, ethyl methyl dimethoxysilane, and disobutyl dimethoxysilane but about said cross linking agent. Preferably it is alkoxysilane, this Silan has methyl trimethoxysilane, and its methyl triethoxysilane, and more preferred methyl triethoxysilane, and the most preferred.

[0061] As for a hydrolytic basis on Slant, it is preferred that it is the same organic reagent as a hydrolytic basis on a cross linking agent. Therefore, as for a hydrolytic basis on Slant, when a hydrolytic basis on a cross linking agent is a methoxy group, it is preferred that it is an alkoxy group. But they do not necessarily need to be methoxy groups. Combination of two or more leaving groups which may give dangers of immiscible nature or exothermic acid / base reaction should be avoided.

[0062] If ingredient (II) and ingredient (III) are mixed, while incorporated about an amine, functionality of ingredient (II) and ingredient (III) will have been

Protein- α -dextrin (III) and (IV) are mixed by various mode ratios, and an amino functionality viscosity above 1000 cP is the increase 7–28 days, gelling will take place, without viscosity of an amino functionality siloxane is the increase 10 opposite ingredient (II) and adding making into less than one a mole ratio which is ingredient (II) opposite ingredient (II) and adding ingredient (IV). It depends for this actual time interval on quantity of added amino functionality Siloxane.

silicone is formed. Usually, 1:1, even though there are few mole ratios of ingredient (IV) and (V) opposite ingredient (III), these ingredients are mixed so that it may be preferably set to at least 1:1; and a mole ratio of ingredient (IV) opposite ingredient (II) may be set to 0-2. The most desirable mole ratios of ingredient (IV) opposite ingredient (II) are 0-2-0.8, and the most desirable mole ratio of ingredient (IV) opposite ingredient (III) are 0-2-0.8.

sections is argued as being merely a *“tame”* amino group in an amino functionality siloxane compound, and who of the JUDSON GROUP by a weight/section of 100 weight section, on the basis of JDUSON siloxane polymer [0010] for said amino functionality siloxane, from start of emulsification until when being added after an amino functionality siloxane emulsifying ensures that the adhesive property of a silicone elastomer obtained when water is removed does not decrease, it is 2 to 4 hours preferably. Adding an amino functionality siloxane within 8 hours after emulsification 1, and more preferably, it is preferred to add before emulsifying an amino functionality siloxane for an ease of combination.

[0016] Ingredient (G) is acidic and thus is used as an optional component. Acid is believed to help in an amino functionality siloxane to enter into an emulsion. As another ingredient, this acid can be added as a part of other ingredients. For example, acidic agent which was formed when this DBTDA is heated/combined with acid, when used with a solution when diisobutyl dicarboxylate (DBTDA) is used.

as a condensation catalyst, or adding to a drainage system emulsion. In this invention, $\text{HCl} \cdot \text{H}_2\text{SO}_4$, organic acid, carboxylic acid, can use any acid, for example, Carbonyl acid is a preference. That is because for carboxylic acid and those corresponding amine salt act also as condensation catalyst with a tin condensation catalyst. As an example of carbonyl acid, there are acetic acid, formic acid, propionic acid, and crotonic acid. Acetic acid is the most desirable acid. Since orthobenzoic acid is the most desirable acid. Since orthobenzoic acid is the most desirable acid. When carboxylic acid is added, quantity acid and those corresponding amine salt act as condensates, when carboxylic acid is added, quantity a tin condensation catalyst can be reduced.

[0068] Usually, acid can apply quantity of 0.05 to 0.3 weight section on the basis of siloxane polymer 100 weight section. Preferably, 0.06 to 0.2 weight section is added on the basis of siloxane polymer 100 weight section, and this acid has 0.07 to 0.13 much more preferred weight section.

[0069] Silicic acid is added as a single kind or a mixture of a kind beyond 2 or it. This acid can add a front, startup of emulsification as a behind, A. Adding is preferred before it, this acid has before a bridge formation of JURQ/GANO siloxane polymer therefore a condensation catalyst, and a required cross linking agent.

1500-18000 A DETAILED DESCRIPTION

[0015] Since the performance characteristics of a silicone elastomer formed from a silicone emulsion or oil or if a certain kind is arrested, additional arbitrary ingredients, for example, a filler, and other ingredients may be added with other ingredients according to a wish. As an example of a reinforcing filler and a filler for increase in quantity, there are calcium carbonate, a titanium dioxide, zinc oxide, iron oxide, and kaolin clay. An example of a filler which can be used in order to give a retardancy or adhesion are resinates, there are silicon 3 hydrate, a bentonite, and wary acid. Other arbitrary ingredients which include paints, stabilizer, or reinforcing nature resin in the spot can be added to this invention silicone emulsion.

atmosphere at a room temperature. The polymerization can be carried out in a closed system. The polymerization time depends on the type of the cross linking agent used. For example, the polymerization time is 1 to 24 hours for $\text{[MMA]} = 25 \text{--} 80 \text{ wt.}\%$, generally time of a polymerization is 1 to 24 hours depending on temperature and a molecular weight of a *wet* or *dry* polymer. After *JURIGON* siloxane polymer reaches a molecular weight of a wish, a polymerization is suspended by introducing this monomer.

[0077] At least one amid functionality siloxane is added at any time between emulsion polymerizations; for example, a reserve mixture of some reserve mixtures before emulsification is conducted, a time after the start up of a polymerization, is added behind A for 2, when being added before said amino functionality siloxane's emulsifying, adding within 8 hours is preferred. [0078] [0079] comparatively low concentration polymer solid content is desired water of an addition amount may be added at which stage of combination. A general polymer solid content is 20 to 75%. A desirable polymer solid content is 40 to 75%. A more preferable polymer solid content is 45 to 75%.

[0079] This acid may be independently added as a part or outer coating, such as a polymer or emulsion, as a part of other ingredients. DBTB will form acidic ester, for example, when it adds to water.

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http://www4.ipm.ac.ir/all.html#all2&cat=4&row=1&col=1

nominic surface active agent is used as independent or a mixture. This mixed device is well-known as a person skilled in the commercial mining device of one of types. [084] A cross linking agent (when required, or when it is wished) and the ion condensation catalyst can add a front strum of emulsification after emulsification. However, said cross linking agent and the ion condensation catalyst will often be added before emulsification. It is said after emulsification that the cross linking agent must sink into a disperse phase from water, and must still maintain that recyclability.

[0088] When a polymer solid content is cleared, watch or an addition amount can also be added within 8 hours after bridge construction or an exhaustion.

[0089] When a polymer solid content is cleared, a desirable polymer solid content is 10 to 95%. A desirable polymer solid content is one stage of the combination. A desirable polymer solid content is 20 to 85%, and is much more desirable. [40 to 85% of]

[0090] [0087] Acid can also be added at any time as a part of other another or ingredients [name], DDDT/ [0088] will form acidic salt at the time of water, for example, if a desirable method, carbonyllic acid and an amino functionality siloxane are added before bridge construction. If this waters, it will make it possible to reduce quantity of an in condensation catalyst.

[0091] Any arbitrary ingredients can add a filler, paint, stabilizer, resin for reinforcement in the spot or in areas of combination, for example.

[0089] In a more detailed method, it is JURIGANO siloxane polymer (hereinafter referred to as "siloxane polymer") which has a methyl group, respectively — an amino functionality. It is hydroxyl, n is 2, and Y is a methyl group, respectively — an amino functionality. The siloxane of dibutylin dicarboxate catalyst-1 weight section of polyoxyalkylene (alkylene) functionality Silang (INCH₃O)_nSi-agent (OH)₂ CO weight section [water] copy 1 (this). Amino functionality Silang (INCH₃O)_nSi(OH)₂ -NH-(CH₂)₃-NH-(CH₂)₃-NH₂. Hydroxyl and INCH₃O_nSi(OH)₂ b -OH (hereinafter referred to as "siloxane polymer") formed by making Silang (IV) methyl trimellitylamine react, and b is 7-9. And an amino functional group of 0.005 weight sections exists, and a mode ratio of ingredient (IV) and ingredient (V) opposite ingredient (IV) is at least again. A mode ratio of ingredient (IV) part ingredient (IV) and ingredient (V) and end acetic acid of 0.40 copolymer under high shearing, and form high solid concentration emulsion. Subsequently, by adding a bridge in this emulsion, a drainage system silicone emulsion is formed by adding an emulsifier ATOPHETOCO-100 across linking agent of 0.1 weight sections.

[0059] After removing water, a silicone elastomer formed from this invention silicone emulsion has long storage improved adhesive property to a substrate. A silicone emulsion of this invention has long storage compared with the conventional emulsion.

[0091] [Example] The following examples are shown in order to explain the constituent of this invention further. In the following example, unless it is referred in particular, one day after making an emulsion, the cast of the humid emulsion was carried out to the film, and it examined after drying these filmings for seven days.

[0092] [The result of the durometer was obtained by the method indicated to ASTM C681 "IndentationHardness of Elastomeric-Type Sealants Means of a Durometer." The result of the tension and elongation, The size of L, the dumbbell specimen which is 1.27 mm, obtained by the method indicated to "Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers Tension," which uses and was indicated to ASTM D412 The Shore A hardness value was obtained by the method indicated to ASTM C861 "IndentationHardness of Elastomeric Type Sealants by Means

http://www4.ipdl.inpit.gov.in/cgi-bin/tran_web.cgi.cgi?atw_ur=http%3A%2F%2Fwww4.ipdl.i... 2010/04

1000 1000 [DETAIL ED DESCRIPTION]

of Durrometer." **"E"** used here means methyl, "**Et**" means ethyl, and "laboratory conditions" is temperature $\pm 2^\circ\text{C}$, relative humidity $\pm 5\%$ of meanings of **CS 244**.

polymer of 50 Pa⁻¹, and one copy of amino functionality siloxane (AFT2) prepared in Example 1, and stirred it for 30 seconds under the vacuum. Subsequently, DBTDL at 0.06 copy was added and this mixture was stirred for 30 more seconds under the vacuum. Glacial acetic acid of 0.1 copy was added and this mixture was stirred for 30 more seconds under the vacuum. Next, Tergitol(trademark) TMN-

[0084] (A) The adhesive vapour man sample was tonic on its side and had a length of 50 mm in length, and 15 mm in width. The adhesive property was evaluated by putting slitting around it a head using a laser head by sealant / substrate interface, and pulling this head by hand at the angle of 45 degrees to a level base material surface. When a head caused cohesive failure, the adhesive property was graded "easily". The head caused adhesive failure, and when power equivalent to the time of removing a head from a base material surface was required, the adhesive property was graded with good. When adhesive failure was caused by the explosion power in which a head is

comparatively low, it greed with **.

Par (n = 25 **). The 10-L Turculo TM mixer was loaded with the Terpoly(tetradecyltrimethyl ammonium bromide) (TNT) disionization agent (0.1 M), water of 6 L (ethylene glycol monomethyl ether), and 100 copies of (D) of 100 copies.

(D) This mixer was stirred under the vacuum for 5 minutes using the distributed braid revolving at 1500 rpm. After mixing, when it observes, it is transparent and is the gel (CHIKUSOTO type) of non ionogenic.

these addition. The gel of emulsion mixed it; it was and now this part was removed by a lump nor gel. To this emulsion, the hydroxyl and POMs (the degree of polymerization = 6) viscosity of 25 ± 0.4 Pa·s at 76 °C copies, the N-(2-aminomethyl)-N-methacryloyl, and the methyl TORIME TSU sian (MTO) of 10.2 copies. The obtained amino functionality silane (AFeZ) of 50 copies was added by carrying out cold vacuum. The distributed braid was rotated at 600 rpm, stirring of this mixture was started. After this mixture was applied, and this mixture was stirred for 2 minutes, subsequently — adding BTMs,

(isobutyl trimechomethyl) or 40 copies — the bottom of a vacuum — a mixture — further — it was stirred for 2 minutes. Next, DTS/TA (dibutyl sebacate) of ten copies was added and this mixture was stirred for 2 minutes using the distributed braid rotated at 600 rpm under a vacuum. The Semco (air cartridge) cartridge was filled up with this silicone emulsion that constructed the bridge, and the air which centrifuged it was caught was removed.

needs to harden under a laboratory condition for seven days. The tensile strength of this hardened translucent silicone elastomer was 0.5MPa (20 Pa), 200% of the maximum elongation, and the Shore A durometer value on the substrate with which it differed, other samples of this aged silicone emulsion were formed to a band 50 mm in length, and 15 mm in width and will be stiffened under laboratory conditions for seven days. Available to one commercial target because of comparison] Calcium carbonate. The latex sealant (Dow Corning trademark) Silicone Plus and the RTV alkoxy [Ceramco Electro-Feldmark GE] of one commercially available

hydroscopic-porous moisture permeability (water-resist) with which it was filled up. It evaluated under the same condition. Table 1 shows an adhesive result.

(alkyd paint). A. An oil paint spreading prime (unat plasma). B. An oil paint spreading prime (unat plasma). C. Good A. Glass A. Good A. Brass A. A. aluminum. D. A. Good A. PVA. E. Good A. PVA. F. Good A. Mortar A. Good Right PMMA A. A. A. polyacrylate ** Good ** stainless steel A. A. Right mortar A. Good Right PMMA (polydimethylmethacrylate) ** Good ** [00105] [Example 1].
(a). The viscosity in 25 °C is 2500 cP. Load the White Mix™ pot of 350mL with 100 copies of hydroxy end POM.

http://www4.ipdl.nipk.go.jp/cgi-bin/tran_wes.cgi?S1=1

JP.09-183902.A [DETAILED DESCRIPTION]

polymer of 50 Pa⁻¹, and one copy of amino functionality siloxane (AFT2) prepared in Example 1, and stirred it for 30 seconds under the vacuum. Subsequently, DBTDL at 0.06 copy was added and this mixture was stirred for 30 more seconds under the vacuum. Glacial acetic acid of 0.1 copy was added and this mixture was stirred for 30 more seconds under the vacuum. Next, Tergitol(trademark) TIN-

Glossary

Figure 14 (Example B) AFMPTMS of the molar quantity used about AFF2 in Example A in the poly(ethylene terephthalate) matrix. By carrying out cold blending of siloxane diol (viscosity 0.04 Pa·s at 25 °C) and siloxane diol (TEOS-9, or two more amino functionality, alkoxanes were prepared, using the following scheme:

[0125] [Table 8]
The table shows functionality, siloxane content, (part) AFF2/FF11, siloxane d10 DP=84.6
71.59/68.07/AEPTMS 14.59/13.71/TM 1.02 – TEOS – 18.02/01/16/AFF2,
AFF2 and AFF3 were evaluated in the siloxane composition, respectively. In each case, the
addition of AFF2, AFF12 or AFF13 was kept constant as one copy on the basis of 100 copies of
PDMS polymer. As it was prepared and filled up with these compounds and had indicated for Example
4, it is examined about the adhesive property. All the blended sample showed cohesive failure mode
of adhesion. These adhesions were examined also about freeze thawability, and it found our possess in

[01] (21) Example 9) Molar quantities of the molar quantity used about AF2 in Example 4 in the polyethylene container. By carrying out cold blending of viscose diol (Viscose 0.04 P = 25 °w/w) and the alkoxyl functionalized siloxane were prepared : using the OLFUNOMI state of DP = 7-9, two or more amino functionality siloxane — MTM, tetraethyl orthosilicate (TEOS), and propyl orthosilicate (NPOS). Following alkoxysilylation — MTM, tetraethyl orthosilicate (TEOS), and propyl orthosilicate (NPOS). This alkoxyl OLFUNOMI silane mixture 35 of tetra (methylsilyloxy) ethoxy silane (TOS) 84% tri- and dioxy MOSHANM silane 10% of mono- and dioxy MOSHANM silane on a mole basis on a molecule are ethoxy

bases here), and 2% of methylated keto KISHIMO silane solvent were included. This mixture was made to react for 24 hours, and when measured by GC after 100% of OH groups of silanone had disappeared, AFT2 was prepared. The 6th table shows the presentation of the prepared amino functionality siloxane.

アミノ官能性 シロキサン化合物	ANF 2.	ANF 1.4
シロキサンゴループ SHO-CH ₂ -CH ₂ -NHR	74.4	67.8

AEAPTMS	15.2	13.83
MTM	10.2	-
TOS	-	18.19

[0129] AFT2 and AFT4 were evaluated in the elastomeric composition, respectively, in each case. The addition of AFT2 or AFT4 was kept constant on the basis of 100 copies of PDMS in the polymer. As it was prepared and filled up with these compounds and had indicated for Example 4, it examined about the cohesive property. All the blended samples showed cohesive failure mode to gases. These were examined also about freeze thaw stability and it found out passing in 10 times of ATSM *** cycles.

react for 24 hours, and the effect of the 2 premix time of two or more ingredients containing an amine functionality, siloxane was evaluated.

- What blended AEPMTMS with siloxane diol (DP 7-9, 25 ** viscosity 0.04 Pa \cdot s);
- What blended AEPMTMS with siloxane diol (DP 7-9, 25 *** viscosity 0 Pa \cdot s);
- MTM.

[0131] [The 9th table shows the presentation of prepared various functional fluids.
 [0132] **[Table 10]**
 The 9th table official Ability Sex Style: 16 ingredients (part) of body AFF15 F1 AFF(c)(a), (b), (c)
 skatole dol DP-12, 07 37.97 -TEAPMTM-16.93 - 50 SA4NTM-1 - 12.03 40.16[0133] GLC analysis
 of skatole dol DP-12, 07 37.97 -TEAPMTM-16.93 - 50 SA4NTM-1 (b) had reached. Three compounds
 said [functional fluid showed that] MTM and siloxane dol of (b) had not reached. These compounds
 which one of said the functional fluids, respectively were prepared and evaluated.
 [0134] A mixture obtained about ascorbic acid compound by
 PDA Pro-6 in 200 copies. A total (rademan) TMN-6 surface active agent of four copies.
 [0135] In 25 kg of

[Table 11] adhesive property to compound, nature S-AFF15 S-F1 S-AFF15 glass of the 10th table sealant, Condensation Condensation a condensation diuron (Shira A) — 6 12 Tensile strength (MPa) — 0.47-0.65 The 0.55 maximum elongation (%) 1000 880 1280 in order to measure the stability various functionals, the sample of each 15-g fluid was put into the polyethylene bottle, and elastomers are shown in the 10th table.

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bases here), and 2% of methylated keto KISHIMO silane solvent were included. This mixture was made to react for 24 hours, and when measured by GC after 100% of OH groups of silanone had disappeared, AFT2 was prepared. The 6th table shows the presentation of the prepared amino functionality siloxane.

アミノ官能性 シロキサン化合物	ANF 2.	ANF 1.4
シロキサンゴループ SHO-CH ₂ -CH ₂ -NHR	74.4	67.8

AEAPTMS	15.2	13.83
MTM	10.2	-
TOS	-	18.19

[0129] AFT2 and AFT4 were evaluated in the elastomeric composition, respectively, in each case. The addition of AFT2 or AFT4 was kept constant on the basis of 100 copies of PDMS in the polymer. As it was prepared and filled up with these compounds and had indicated for Example 4, it examined about the cohesive property. All the blended samples showed cohesive failure mode to gases. These were examined also about freeze thaw stability and it found out passing in 10 times of ATSM *** cycles.

react for 24 hours, and the effect of the 2 premix time of two or more ingredients containing an amine functionality, siloxane was evaluated.

- What blended AEPMTMS with siloxane diol (DP 7-9, 25 ** viscosity 0.04 Pa \cdot s);
- What blended AEPMTMS with siloxane diol (DP 7-9, 25 *** viscosity 0 Pa \cdot s);
- MTM.

[0131] [The 9th table shows the presentation of prepared various functional fluids.
 [0132] **[Table 10]**
 The 9th table official Ability Sex Style: 16 ingredients (part) of body AFF15 F1 AFF(c)(a), (b), (c)
 skatole dol DP-12, 07 37.97 -TEAPMTM-16.93 - 50 SA4NTM-1 - 12.03 40.16[0133]GLC analysis
 of skatole dol DP-12, 07 37.97 -TEAPMTM-16.93 - 50 SA4NTM-1(d) had reached. Three compounds
 said [functional fluid showed that] MTM and siloxane dol of (b) had not reached. These compounds
 which one of said the functional fluids, respectively were prepared and evaluated.
 [0134] [The 9th table obtained about ascorbic acid compound by
 PDA Pro-6 in 200 copies. A total of (rademan) TMN-6 surface active agent of four copies.
 [0135] [The 9th table shows the presentation of prepared various functional fluids.

[Table 11] adhesive property to compound, nature S-AFF15 S-F1 S-AFF15 glass of the 10th table sealant, Condensation Condensation a condensation durometer (Shore A) → 6–12 Tensile strength (MPa) → 0.47–0.65 The 0.5% maximum elongation (%): 1000 880 1280 in order to measure the stability various functionals fluids, the sample of each 15 g fluid was put into the polyethylene bottle, and elastomers are shown in the 10th table.

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poured with nitrogen, and stored into a 50 ** furnace. AFF15 amino functionality siloxane prepared without using MTM was sealed within four weeks of storage. About F1 and AFF18, no gelling also took place to the same storage time.

[Table 12] Example 11. In the polyethylene container, cold blending of siloxane diol (viscosity 0.04 Pa·s [0.37], M_n, and the functionality) ORGANO siloxane of DP = 7-8 was carried out, and the amine functionality siloxane was prepared by taking this mixture react for 2-4 hours. (a) EA/TMFS which uses the following functionality ORGANO siloxane, (b) Gamma-ethylacryloxypropyltriethoxysilane (GPMTS), (c) gamma-methacryloxypropyltriethoxysilane (MAPTS), and (d) Gamma-methacryloxypropylmethacrylate (MPMTS) were used. Various functionality ORGANO siloxanes were added to said cold blend by the fixed mole ratio. Table 11 shows the presentation of various functional fluids. [0138]

[Table 12] Example 14. Ingredients (part) of the 11th table amino functionality siloxanes, AFF (a) (b) (c) (d) Siloxane No. DP=8 T4.6 74.6 74.8EA/PTMFS = 16.2 - - - MATMS = 17 - - - MFTMS = 13.4MATM, 10.2 1.2 10.2 10.2/10.3[Each of these functional fluid was evaluated in the elastomeric composition. The addition to the silicone latex compound of a functional fluid was kept constant as one copy on basis 100 copies of PDMS polymer. As it was prepared, and filled up with these compounds and had indicated for Example 4, it examined about the adhesive property. The sample using AFF2 (a) showed cohesive failure to glass, and the sample prepared using F2 (b), F3 (c), or F4 (d) showed cohesive failure to glass.

[0140] Example 12. In the polyethylene container, two or more amino functionality siloxanes were prepared by taking cut cold blending of AMTMS, MTM, and the hydroxy and ORGANO siloxane. DP = 7-8 indicated for the example 1 of (a) using the following siloxane diol, the PDMS ORGANOCO siloxane which has viscosity 0.14 Pa·s in 25 **, (b) an average of 1 of average DP = 7-8 and - - - an average of 1 of the polyhydroxyl NECHIRUSHIKIYU ORGANO siloxane of 55% of OH content and (c) average DP = 7 - - - and - - - the PORTOFERURIDORO propylmethyl ORGANO siloxane of 6.10% of OH content. These mixtures were made to react for 24 hours, and when measured by GLC after that, 100% of -OH functional groups of the hydroxy end ORGANO siloxanes had disappeared. The 12th table shows the presentation of prepared various amino functionality siloxanes.

[Table 13] Example component (part) AFF2AFF7/ AFF18 dimethylsiloxane did 74.6 12th table amino functionality siloxane component (part) AFF2AFF7/ AFF18 dimethylsiloxane did 74.6 - - - phenylmethoxy siloxane diol = 14.77 - - - trifluoropropylmethyldiisopropylsiloxane = 66.38A/APTMS 115.2 21.08 2012MTH 10.2 14.5 10.1/42[Each of these amino functionality siloxane was evaluated in the elastomeric composition. The addition to said compound of an amino functionality siloxane was kept constant as one copy on the basis of 100 copies of PDMS polymer. As it was prepared, and filled up with these compounds and had indicated for Example 4, it examined about the adhesive property. The sealant sample prepared using (A) showed cohesive failure to glass, wood and concrete, and showed limited destructive mode (50% of cohesive failure) to aluminum and mill finishing aluminum, and prepared using AFF17 or AFF18 showed adhesive failure to glass and mill finishing aluminum, and showed cohesive failure to wood and concrete. The physical properties of all the samples are dramatically alike about the durometer, the Shore range of 4-5 is shown, and tensile strength is 0.34 - 0.37MPa.

The maximum elongation was 200%/ 2300%.

[0143]Example 13]

(a) The following experiments were conducted in order to prove the stability of the reserve mixture which consists of an amino functionality siloxane (AFF2), BTMS, hydroxy end, J/ORGANO siloxane polymer, and DBTDA. The 10.1 Tunisia M mixed was loaded with 500 copy hydroxy end PDMS polymer, ** viscosity 50 Pa·s] and AFF2 of 50 copies which prepared said reserve mixture as follows. This mixture was stirred for 30 seconds under this vacuum, adding 1BTMS of 40 copies, and DBTDA of three copies to this mixture — this mixture — the bottom of a vacuum — further — it stirred for 30 seconds. When the viscosity of this mixture was observed, there was no big increase in viscosity for 4 hours. 10% of this reserve mixture was taken at intervals of 60 minutes, and Whip Mix™mixer of 350ml was loaded. When Tergitol(trademark) THIN-6 surface-active agent of two

copies and DI water of two copies were added to each of these samples and this mixture was stirred for 30 seconds under this vacuum, it was slightly translucent and the o/w emulsion of high solid content was obtained with non fluidity. Subsequently, DI water was stirred for 30 seconds under the vacuum between each addition [portions / four of four copies each]. The sample taken from 1, 2, 3, and the reserve mixture of four copies after was emulsified spontaneously at all, and the obtained estomene showed cohesive failure to glass.

(b) Other experiments were conducted, in order to evaluate whether the amino functionality siloxane has been added however for a long time after emulsifying. Hydroxy end PDMS polymer (25 ** viscosity 50 Pa·s) of 5000 copies, BTMS of 40 copies, and DBTDA of three copies were added, and this mixture was stirred for 30 seconds under this vacuum. Tergitol(trademark) TMN of 100 copies were added to this mixture, and this mixture was stirred for 30 seconds under the vacuum. Subsequently, when DI water of 150 copies and this mixture was stirred under the vacuum for 5 minutes, it was slightly translucent and the o/w emulsion of high solid content was obtained with non fluidity. Whip Mix™mixer of 350ml was loaded with these samples, and AFF2 of one copy was added. Subsequently, this mixture was stirred for 30 seconds under the vacuum. To this high solid content silicone emulsion, DI water was divided into four portions of four copies, respectively, and it added, and stirred for 30 seconds under the vacuum between each addition. The amino functionality siloxane could be added a little emulsification of other ingredients of till the 8 hours, and, in addition, giving the cohesive failure of the hardened sealant to glass was found out.

[Table 12] Example 14. Whip Mix™pot of 350ml, was loaded with hydroxy end and PDMS polymer (viscosity of 25 ** [0144]) Whip Mix™pot of 350ml, and DBTDA of 0.06 copy, and this mixture was stirred for 30 seconds under the vacuum, adding AFF2 of 0.93 copy to this mixture — the bottom of a vacuum — further — it stirred for 30 seconds under the vacuum, adding diacidic acidic acid of 0.08 copy — this mixture — the bottom of a vacuum — further — it stirred for 30 seconds under the vacuum — further — it stirred for 30 seconds subsequently — adding diacidic acidic acid of 0.07 copy was added to this mixture, and this mixture was stirred for 30 seconds under the vacuum. adding Tergitol(trademark) TMN-6 and DI water of 2.32 copies of 0.06 copies to this mixture — this mixture — the bottom of a vacuum — further — when stirred for 30 seconds, it was slightly transparent and the o/w emulsion of high solid content was obtained with non fluidity, adding 66.29 copies of underwater kaolin viscosity dispersing elements of 70% of solid content, and 44.9 copies of dispersing elements of the underwater kaolin viscosity dispersing elements of 70% of solid content to this mixture — this mixture — the bottom of a vacuum — further — it stirred for 30 seconds. All the air that filled up the Senco (trademark) cartridge with this silicone emulsion, centrifuged, and was caught was removed. After aging this emulsion at a room temperature for 1 hours, the sample of each combination was formed on the 2.54 mm (100 mils)-brick film, and it was made to hardened under a laboratory condition for 14 days. In tensile strength, 0.84MPa (82 psi) and the maximum elongation were [640% and the Shore A durometer of this silicone emulsion is 22. Furthermore it aged for 18 hours under a laboratory condition for 14 days. This material showed the adhesive property (cohesive under a laboratory condition for 14 days. This material showed the adhesive property (cohesive failure mode) excellent in all the substrates.

[Translation done.]